

## **REMARKS**

Favorable reconsideration is respectfully requested in view of the foregoing amendments and the following remarks.

### **I. CLAIM STATUS & AMENDMENTS**

In items 4 and 6 on page 1 of the Office Action, claim 20 was erroneously indicated as pending and rejected. However, claim 20 was cancelled in the response of January 22, 2004.

Accordingly, claims 1, 2, 4-12, 14-19, and 21-23 were pending when last examined.

Claims 15-17 are withdrawn as non-elected subject matter.

Claims 1, 2, 4-12, 14, and 18-23 have been examined on the merits, and stand rejected.

Claims 1 and 18 have been amended to recite "laser abration" instead of "laser abrasion" thereby better clarifying the claimed invention. Similarly, the specification at page 5, lines 27-31, at page 6, lines 13-18, and at page 7, lines 25-30 has also been amended to recite "laser abration" instead of "laser abrasion." Laser abration is a technique commonly used in the field. Support for this language can be found throughout the Specification, for instance, the methodology for film formation described at page 6, lines 13-22 is more appropriately known and referred to as "laser abration."

Also enclosed is an attachment sheet for Figures 1 and 2 that corrects a typographical error with regard to the numbering of "21" and "22" in Figure 2. In new Figure 2, "21" correctly corresponds to the target and "22" correctly corresponds to the substrate. Support for this amendment can be found in the specification at page 7, lines 17-24, wherein it is indicated that the substrate corresponds to "22" and the target corresponds to "21" for Figure 2. In

Therefore, no new matter has been added by this amendment.

### **II. REJECTION UNDER 35 U.S.C. § 103(A)**

#### **A. Mikio in view of Araki (claims 1,-4, 7, and 8-14)**

Claims 1-4, 7, and 8-14 are rejected under 35 U.S.C. § 103(a) as obvious over Mikio et al., JP 09-237714 ("Mikio") in view of Araki et al., U.S. Patent No. 5,676,998 ("Araki"). See item 3 on page 2 of the Office Action.

The statement of rejection erroneously indicates that the rejection is under 35 U.S.C. § 102(b), as opposed to § 103(a). Based on the section heading immediately preceding the

rejection and the reasons set forth for the rejection, it is clear that an obviousness rejection under 35 U.S.C. § 103(a) was intended. Accordingly, the rejection has been treated as an obviousness rejection under 35 U.S.C. § 103(a).

This rejection is respectfully traversed for the same reasons discussed in the response dated January 22, 2004 and for the following reasons.

To establish obviousness, three criteria must be met. First, the prior art references must teach or suggest each and every element of the claimed invention. Second, there must be some suggestion or motivation in the references to either modify or combine the reference teachings to arrive at the claimed invention. Third, the prior art must provide a reasonable expectation of success.

In this case, the claims call for a method for manufacturing a rare earth **thick** film magnet of **30-100  $\mu$ m in thickness** and a **film-formation speed of 50  $\mu$ m/hr or more**.

Mikio fails to disclose or suggest each and every element of the claimed invention, namely a rare earth thick film magnet of 30-100  $\mu$ m in thickness. Instead, Mikio discloses a **thin** film magnet for use in motors or magnetic appliances.

The rejection is based on a misunderstanding regarding the instant application's characterization on the page 2, line 9 of Japanese Patent Laid-Open Publications No. 09-237714 (i.e., the Mikio patent application) and No. 11-214219. The instant specification indicates that these Japanese applications disclose a "multi-layer rare earth thin film magnet of 0.01-300  $\mu$ m thick." However, this passage may be misinterpreted in that Mikio never discloses a **thick** film hard magnetism layers with such a thickness of 30-100  $\mu$ m. Instead, Mikio discloses a thin film and not a thick film. This thin film is formed with multiple layers, i.e., 5 times multiplying of 20 nm Nd-Fe-B hard magnetism thin film layer and 20 nm Fe soft magnetism thin film layer. Moreover, the process in Mikio achieves a total thickness of only **200 nm** as evidenced by Examples 1-4 in Mikio. In this sense, the methodology in Mikio **teaches away** from the claimed thickness. It is well established that a teaching away is indicative of non-obviousness. Moreover, given this teaching away, Mikio fails to contain a reasonable expectation of success at modifying the methodology to arrive the claimed rare earth thick film magnet of 30-100  $\mu$ m in thickness.

Also, it is noted that the layer of thickness varies from 15 nm to 50 nm in Mikio. See Examples 6-10 of Mikio. The number of lamination is not disclosed in Mikio. However, the

number of lamination does not drastically change due to the layer formation speed of the process of Mikio, which is done by bipolar magnetron sputtering. Similarly, it is noted that the film of JP 11-214219 was formed by a physical vapor deposition such as sputtering, vacuum deposition and ion plating. See paragraph 0054 in JP 11-214219.

More importantly, in the process of Mikio utilizing bipolar magnetron sputtering, the layer formation speed is 2.0  $\mu\text{m}/\text{hour}$  for Nd-Fe-B and 0.3  $\mu\text{m}/\text{hour}$  for Fe, respectively. Assuming arguendo, that Mikio were to make a 300  $\mu\text{m}$  magnetic layer as required by the claims, or 150  $\mu\text{m}$  of Fe layer is formed, it would take 500 hours (approximately 21 days) to form only one layer, thereby rendering the teaching in Mikio impractical.

On the other hand, Applicants invented a process for manufacturing a thick film magnet with a high  $H_c$  (at least 6-10 kOe or 478-796 kA/m) within a short period of time of 10-60 minutes. Clearly, the claimed invention achieves **surprising and unexpected results** in the manufacture of a practical magnet within 10-60 minutes, as compared to 500 hours as in Mikio. Such surprising and unexpected results are also indicative of non-obviousness.

Moreover, the “thin film magnet” of Mikio and the “thick film magnet” of the present invention are completely different compositions differing in material characteristics, manufacturing processes, and in production costs.

Again, as discussed in the prior response, the thickness of the thin film disclosed in Mikio is 0.02  $\mu\text{m}$  (200 Å) Nd-Fe-B and 0.02  $\mu\text{m}$  Fe. Neither the thickness of the thin film nor the thickness of the laminated thin-film made by laminating a plurality of the repeating unit disclosed in Mikio is thick enough to obtain the sufficient magnetic characteristics as disclosed in the “Background of the Invention” of the present invention which characteristics result from the present method and are present in the claimed motor.

As evidence that a “thin film magnet” and a “thick film magnet” are different compositions, the Examiner is again directed to the definitions of the two types of film as commonly understood by those in the technical field as evidenced by the copy of the McGraw-Hill Dictionary of Scientific and Technical Terms, 6<sup>th</sup> Ed which was attached to the prior response. The phrase “thin film” is defined as a film that is a few molecules thick film and is deposited on a substrate to form a capacitor, resistor, coil, cryotron, or other circuit component. In other words, a film having a thickness in the range of **0.01-1.0  $\mu\text{m}$**  is a “thin film.” By

contrast, the “thick film magnet” of the present invention has a thickness of 30 to 100  $\mu\text{m}$  and would never be considered a “thin film magnet.”

Also, enclosed herewith is a copy of a page from the third edition of IEEE Standard Dictionary of Electrical and Electronic Terms published by the Institute of Electrical and Electronics Engineers. This publication defines the term “magnetic thin film” as “usually less than 10,000 angstroms” which corresponds to 1.0  $\mu\text{m}$  thick.

Thus, the assumption that Mikio discloses a thick film is inaccurate. Mikio simply fails to disclose or suggest a rare earth thick film magnet of 30-100  $\mu\text{m}$  in thickness.

In addition, Mikio fails to teach physical deposition by laser ablation. Also, the deposition rate for “thin films” is conventionally 0.1-4  $\mu\text{m/hr}$ . Given such a slow deposition rate, production costs for conventional thin film process would increase drastically to obtain a sufficient thickness of 30-100  $\mu\text{m}$ , such as disclosed that in the present invention. As discussed in the specification, the present invention provides a less expensive method to form a high performance magnetic film. In this regard, the “thick film magnet” of the present invention is obtained by the method called “laser ablation” and the deposition rate is very high compared with the conventional thin film process.

Also, claim 2 calls for laminating a plurality of thick films, but Mikio fails to teach this. Instead, Mikio discloses the lamination of thin films, not thick films.

Claim 8 calls for a degree of vacuum of  $10^{-6}$  Torr or less. Mikio also fails to disclose this. Instead, Mikio discloses a degree of vacuum of  $8 \times 10$  to 1 Pa at the time of deposition which is equivalent to 0.060 to 0.005 Torr. Before the deposition, the chamber is first evacuated to  $8 \times 10^{-4}$  Pa and then Ar gas is introduced to the chamber to perform the deposition at the above-described vacuum range.

Thus, Mikio also fails to disclose or suggest a film-formation speed of 50  $\mu\text{m/hr}$  or more, the lamination of thick films, and a degree of vacuum of  $10^{-6}$  Torr or less. In sum, Mikio simply fails to teach and/or suggest each and every element of the claimed invention, and the reference lacks a reasonable expectation of successfully achieving a thick film magnet with the requisite thickness.

The secondary reference of Araki fails to remedy the deficiencies of Mikio.

First, as discussed above, the primary reference, Mikio, discloses a thin film magnet, and not a thick film. Araki fails to remedy this deficiency because it does not teach a thick film magnet.

Second, Araki is relied upon as teaching laser ablation as one film formation method to form a magnetic layer along with PVD. While Araki merely recites the possibility of forming a magnetic layer by laser ablation, the reference fails to provide a precise explanation of such process. In other words, Araki's teaching with regard to laser ablation is insufficient to arrive at the claimed invention.

An actual  $R_2Tm_2B$  magnet formed by laser ablation was reported in Yang et al., Journal of Applied Physics, vol. 83, no. 11, pp. 6620-6622 (1998), a copy of which is enclosed. In Yang, a magnet having  $H_c$  of 23.9-79.6 kA/m (i.e., the equivalent to 300-1 kOe) was formed by KrF laser ablation. However, the  $H_c$  value was not sufficient when compared with laser ablation method resulting in an  $H_c$  of 875 kA/m for a SmCo magnet reported in Cadieu et al., Journal of Applied Physics, vol. 83., no. 11, pp. 6247-6249 (1998), a copy of which is also enclosed.

In view of these references, it is evident that Araki fails to teach or suggest the procedure for manufacturing a practical magnet by laser ablation. Figure 3 of Araki is only a general idea of such a method. However, this is insufficient to suggest the method of the claimed invention. Moreover, a comparison of Figure 3 of Araki with Figure 2 of the instant invention reveals that the structures are completely different.

Only the Applicants of the present invention have obtained a process of forming a thick film magnet with a high  $H_c$  (at least 6-10 kOe or 478-796 kA/m) within a short period of time (10-60 minutes). The large  $H_c$  value and short production time can never be achieved by combining Araki with Mikio.

Third, as discussed in the prior response, Araki discloses the following two "important factors" regarding their invention: (1) the substrate temperature should be kept high enough (500-630°C) to obtain a sufficient crystallization, even in a case where the film is post heat-treated (Araki, column 7, lines 33-37); and (2) the deposition rate should be less than 40  $\mu\text{m/hr}$  (Araki, column 8, lines 25-29 and Table 2).

By contrast, in the present invention, substrate heating is not necessary. Moreover, the deposition rate in the claimed invention is 50  $\mu\text{m/hr}$  or more. Araki's disclosure of a deposition

rate of less than 40  $\mu\text{m/hr}$  **teaches away** from the claimed deposition rate. Again, such a teaching away is indicative of non-obviousness.

Thus, Mikio and Araki fail to teach and/or suggest each and every element of the claimed invention. Furthermore, there is no suggestion and/or motivation in either reference to modify/combine the teachings of the two references to arrive at the claimed invention, and the references lack a reasonable expectation of successfully arriving at the claimed invention. Thus, Mikio and Araki cannot render the claimed invention obvious.

Therefore, the rejection of 1-4, 7, and 8-14 under 35 U.S.C. § 103(a) is untenable and should be withdrawn.

**B. Mikio in view of Araki and Akioka (claims 5 and 6)**

Claims 5 and 6 are again rejected under 35 U.S.C. § 103(a), as obvious over Mikio in view of Araki, and further in view of Akioka, U.S. Patent No. 5,597,425 ("Akioka"). See item 4 on page 5.

This rejection is respectfully traversed for the reasons noted immediately above with regard to Mikio and Araki and for the following reasons.

The claims call for a method for manufacturing a rare earth thick film magnet of 30-100  $\mu\text{m}$  in thickness wherein the substrate includes tantalum or ion-implanted tantalum on a surface.

As discussed above, Mikio and Araki fail to disclose or suggest a thick film magnet with the requisite thickness. For this reason alone, these references cannot render the claimed invention unpatentable. Akioka also fails to disclose or suggest a thick film magnet with the requisite thickness.

Thus, Mikio, Araki, and Akioka fail to teach and/or suggest each and every element of the claimed invention. Furthermore, there is no suggestion and/or motivation to combine the reference teachings to arrive at the claimed invention. Thus, the cited references cannot render the claimed invention obvious.

Therefore, the rejection of claims 5 and 6 under 35 U.S.C. § 103(a) is untenable and should be withdrawn.

**C. Mikio in view of Araki and of Bell (claims 18 and 19)**

Claims 18 and 19 stand rejected under 35 U.S.C. § 103(a), as obvious over Mikio in view of Araki and further in view of Bell et al., U.S. Patent No. 5,682,670 ("Bell"). See item 5, on page 8 of the Office Action.

Applicants again respectfully traverse this rejection for the reasons noted above with regard to Mikio and Araki and for the reasons set forth below.

The claims call for a method of manufacturing a rare earth thick film magnet comprising deposition by laser ablation and a substrate that includes tantalum on ion-implanted tantalum on a surface.

Like Mikio and Araki, Bell also fails to disclose or suggest a thick film magnet with the requisite thickness.

Accordingly, Mikio, Araki, and Bell fail to teach and/or suggest each and every element of the claimed invention. Furthermore, there is no suggestion and/or motivation to combine the reference teachings to arrive at the claimed invention. Thus, the cited references cannot render the claimed invention obvious.

Therefore, the rejection of claims 18 and 19 under 35 U.S.C. § 103(a) is untenable and should be withdrawn.

**D. Mikio in view of Bell and Akioka (claims 22 and 23)**

Claims 22 and 23 are again rejected under 35 U.S.C. § 103(a), as obvious over Mikio in view of Bell and Akioka. See item 6 on pages 7-8 of the Office Action.

This rejection is respectfully traversed for the reasons noted above with regard to Mikio, Bell, and Akioka.

Like Mikio and Bell, Akioka also fails to disclose or suggest a thick film magnet with the requisite thickness.

Accordingly, the cited references fail to teach and/or suggest each and every element of the claimed invention. Furthermore, there is no suggestion and/or motivation to combine the reference teachings to arrive at the claimed invention. Thus, the cited references cannot render the claimed invention obvious.

Therefore, the rejection of claim 22 and 23 under 35 U.S.C. § 103(a) is untenable and should be withdrawn.

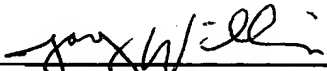
**CONCLUSION**

In view of the foregoing amendments and remarks, that the present application is in condition for allowance and notice to that effect is hereby requested.

If the Examiner has any comments or proposals for expediting prosecution, please contact the undersigned at the telephone number below.

Respectfully submitted,

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**ATTACHMENT TO AMENDMENT AND REPLY**

1. IEEE Standard Dictionary of Electrical and Electronic Terms;
2. C. J. Yang et al., Journal of Applied Physics, vol. 83, no. 11, pp. 6620-6622 (1998);
3. Cadieu et al., Journal of Applied Physics, vol. 83., no. 11, pp. 6247-6249 (1998).

## High coercivity SmCo based films made by pulsed laser deposition

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Films of SmCo based materials exhibiting high intrinsic coercivities and smooth hysteresis loops have been prepared by pulsed laser deposition (PLD) onto moderately heated substrates. Films directly crystallized from SmCo<sub>5</sub> targets onto 375 °C substrates exhibited a maximum  $\mu_0 H_c = 11.3$  kOe at a pulse repetition rate of 10 Hz with lower coercivities for both lower and higher pulse repetition rates. In the present case the films were deposited onto polycrystalline substrates. The films exhibited a very small grain size of less than 1  $\mu\text{m}$  diameter, were mirrorlike, and shadow deposited films were relatively particulate free under scanning electron microscope examination. Shadowed PLD deposition was used for the best films. Laser wavelengths of 193 and 248 nm were used with pulse repetition rates from 5 to 50 Hz. Films grown without shadowing exhibited a great deal of particulate contamination. The hysteresis loops of such nonshadowed films were constricted and exhibited drops in the  $4\pi M$  values upon demagnetization. To our knowledge this is the first reporting of high coercive force SmCo based films deposited by PLD exhibiting single phase type hysteresis loops. © 1998 American Institute of Physics. [S0021-8979(98)42811-1]

### INTRODUCTION

High coercivity films of SmCo based systems have been deposited by sputtering by several distinct variants. Highly textured polycrystalline films have been made by using sputter process control to grow films at thicknesses out to at least 120  $\mu\text{m}$ .<sup>1-4</sup> Such films have the crystalline  $c$  axes nearly randomly aligned onto the substrate plane. Buffer layers are necessary to grow relatively thick films but in principle arbitrarily thick films of highly aligned SmCo deposits can be grown by this method. In contrast to this, highly aligned thin films with thicknesses of less than 0.1  $\mu\text{m}$  have been grown by using substrate film epitaxy.<sup>5,6</sup> The thicker films as grown by sputter process control are generally more suited to device applications. Such relatively thicker films as grown by sputter process control have been used to bias to saturation YIG substrates,<sup>4</sup> to bias permalloy films,<sup>7</sup> and in the construction of a film based magneto-optic waveguide isolator.<sup>8</sup> In this latter device the textured SmCo films were grown directly onto compliant layers directly deposited onto a Bi-YIG optical waveguide so that epitaxy could not have been used to grow such films. Film scale magnetic devices generally employ a magnetically sensitive material such as a magnetoresistive material, a superconducting film element, or a magnetic field sensitive optical material. In addition soft magnetic films are used as flux paths with permanent magnet films used as magnetic biasing elements. Many of the magnetically sensitive types of materials have been readily deposited by pulsed laser deposition (PLD). Such films include high  $T_c$  oxide superconductors, ferrites, and magnetoresistive materials such as the La manganites. It should be noted that these are oxide materials which are generally difficult to sputter deposit with controlled texturing and at appreciable deposition rates. To illustrate the differences in PLD and sputter deposition we have made SmCo based magnetic films by PLD. Although the time averaged deposition rates of

sputter and PLD deposited films are comparable, material is ejected in PLD only during a series of pulses of very short time duration compared to the time between pulses.<sup>9</sup> This means that the relative mobility of deposited surface atoms is very much less for PLD than for sputtering since a complete monolayer can be deposited per pulse. The growth of PLD deposited permanent magnet films also has direct impact on the fabrication of film scale magnetic devices. There have been only a few reportings of rare earth transition metal films as grown by PLD and mostly confined to Nd<sub>2</sub>Fe<sub>14</sub>B.<sup>10</sup>

### EXPERIMENT

PLD, utilizing a Lambda Physik 305Fi excimer laser, has been used to deposit SmCo based films from a set of bulk compound targets. Films have been deposited using wavelengths of 248 and 193 nm, pulse energies of 500–650 mJ at 5–50 Hz with an estimated pulse width of 15 ns. A shadow mask has been used to shield part of the substrate during the PLD process. In this manner the magnetic properties, as well as the number of particulates reaching the substrate in and out of the shadow region, could be observed. Most of the films discussed here have been grown using a substrate temperature ranging from 100 to 750 °C in argon pressures from 100 to 240 mTorr. Film compositions were determined using electron excited x-ray analysis in a scanning electron microscope (SEM). The film composition measurements were calibrated against known bulk composition standards.

### RESULTS AND DISCUSSION

SmCo based films deposited by PLD in the absence of any shadowing onto heated alumina substrates exhibited a strong dependence of Sm concentration on the argon background gas pressure in the deposition chamber. Figure 1 shows the Sm concentration for a series of films deposited onto polished alumina substrates at 375 °C from SmCo<sub>5</sub> targets, and at 500 °C from SmCo "2-17" targets, as a func-

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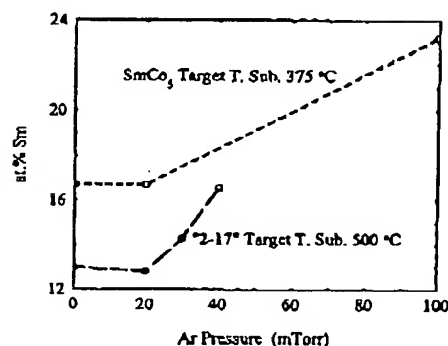


FIG. 1. The Sm at % for a series of PLD films made on to 375 °C substrates from bulk SmCo<sub>3</sub> targets, and films made on to 500 °C substrates from bulk SmCo "2-17" targets, for different Ar pressures during the deposition is shown.

tion of Ar gas pressure. The targets in the latter case consisted of normal bulk type "2-17" magnet material with a composition of Sm 13 at %, Co 58%, Fe 20%, Cu 7%, and Zr 2%. Under vacuum conditions the film Sm concentration corresponded to that of the targets. Films made under vacuum, however, exhibited a large number of particulates causing such films to be unattractive for film device applications. During sputtering large concentration changes can also be effected by using increased sputtering gas pressures and in particular admixtures of Xe as a part of the sputtering gas.<sup>2</sup> Changes in the sputtering gas pressure could be used to vary the film composition from 12 to 18 at %.<sup>11</sup> Higher pressures tend to preferentially scatter the lower mass transition metal atom components relative to the more massive Sm atoms. The net effect is that the lighter transition metal atoms are on the average scattered through larger angles and effectively removed from the deposition beam. Higher pressures in the deposition chamber tend to enrich the Sm concentration in the films.

Room temperature hysteresis loops measured in-plane and perpendicular to the plane are shown in Fig. 2 for a shadow region film deposited at 375 °C and 100 mTorr Ar at a laser wavelength of 193 nm with a pulse rate of 14 Hz. The PLD target was bulk SmCo<sub>3</sub>. The in plane  $H_c$  was 9.7 kOe and the loop shape was characteristic of single phase material. It should be noted that there is no retracing of the in plane hysteresis loop to the highest measurement magnetic field of 18 kOe. An x-ray diffraction pattern for the film of Fig. 2 is shown in Fig. 3. The evident CaCu<sub>5</sub>-type structure (110) dominant texturing and (111) secondary CaCu<sub>5</sub>-type shoulder are consistent with the relative shapes of the in plane and perpendicular to the plane hysteresis loops.

Films of SmCo based materials made without the use of a shadow mask exhibited an appreciable density of particulates on the substrate. Films made in vacuum,  $1.8 \times 10^{-7}$  Torr, on to 550 °C substrates exhibited a small grain size of 0.17–0.30  $\mu\text{m}$  diameter. The particulates tended to be of two principal sizes,  $\approx 0.33 \mu\text{m}$  diameter for the small ones, and  $\approx 0.83 \mu\text{m}$  diameter for the larger ones. The particulate density for nonshadowed films increased with pressure even though the number density of the larger sized par-

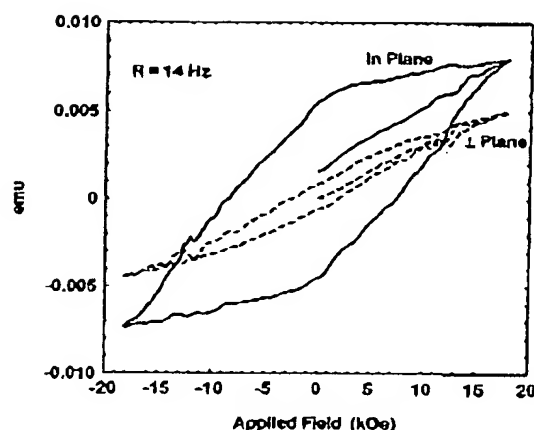


FIG. 2. Room temperature hysteresis loops for a shadow deposited PLD film made from SmCo<sub>3</sub> target at 375 °C on to alumina substrate, pressure 100 mTorr Ar, pulse rate 14 Hz are shown. The key items to note are that the intrinsic coercivity is 9.7 kOe and that the loop shape is smooth.

ticulates remained nearly constant. Nonshadowed films made at 500 °C and 30 mTorr Ar exhibited an average particulate density of  $0.21/\mu\text{m}^2$ , similar films except at 75 mTorr Ar exhibited  $0.33/\mu\text{m}^2$ . In order to limit the number of particulates arriving at the substrate a stainless steel sheet metal shadow was located in the deposition plume to block the direct transport of particulates to the substrate. A higher background gas pressure of argon was then required to scatter the atoms to reach the substrate. The shadow mask strip was arranged midway between the target and substrate so that the shadow blocked a  $13 \times 50$  mm substrate region.

Figure 4 shows hysteresis loops for a SmCo based film shadow deposited at a substrate temperature of 375 °C in 100 mTorr Ar at a laser pulse rate of 10 Hz. The in film plane  $H_c$  was 11.3 kOe. The film surface was mirrorlike which is different from directly crystallized textured SmCo films grown onto alumina films by sputtering. The smooth mirrorlike surface was consistent with a small average grain size. The

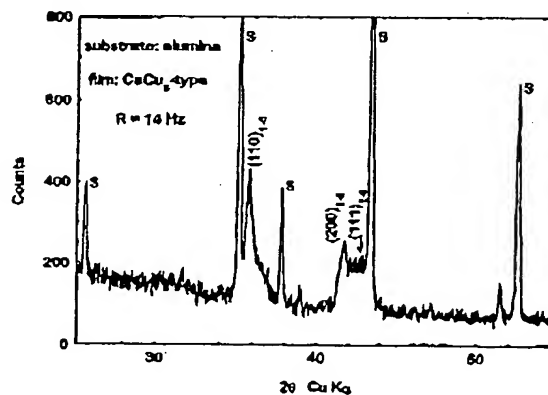


FIG. 3. An x-ray diffraction trace for the film of Fig. 2 is shown which indicates (110) dominant and (111) secondary texturing. The x ray is indexed as CaCu<sub>5</sub>-type structure film. The substrate lines from the polycrystalline Al<sub>2</sub>O<sub>3</sub> are indicated by S in the figure.

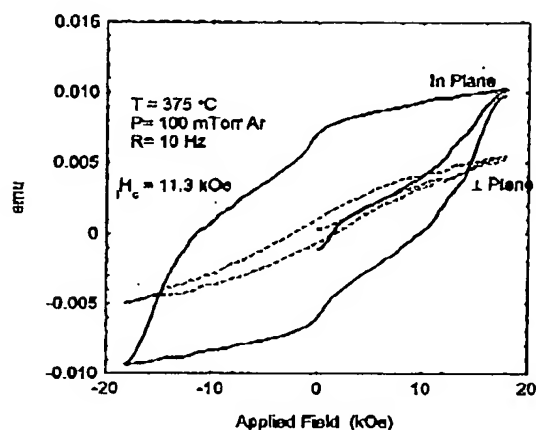


FIG. 4. Room temperature hysteresis loops are shown for a PLD film deposited from bulk  $\text{SmCo}_5$  target at  $375^\circ\text{C}$ , pressure 100 mTorr Ar, shadowed deposition. The laser settings were 193 nm, 600 mJ pulses, 10 ns pulse width, and 10 Hz.

shadow deposited films replicated polish marks on the underlying substrate that made it difficult to determine a film grain size. The deposit in the shadow region was particulate free.

To study the effects of surface atom mobility a series of films were deposited from bulk  $\text{SmCo}_5$  targets in 100 mTorr Ar onto substrates held at  $375^\circ\text{C}$  using  $\lambda=193$  nm for differing pulse repetition rates from 5 to 50 Hz. This is shown in Fig. 5 where the  $H_c$  values versus laser pulse rate are shown for a series of films shadow deposited under the same conditions except for the laser pulse repetition rate. The higher pulse rates result in low coercivities due to insufficient time between pulses for surface atom site adjustments.

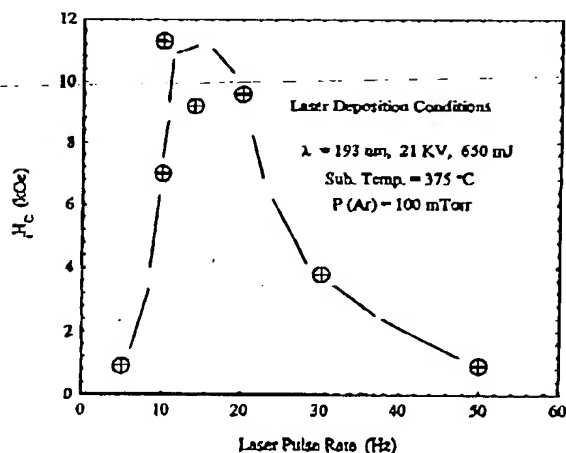


FIG. 5. The coercivity  $H_c$  vs laser pulse rate is shown for a series of films shadow deposited from  $\text{SmCo}_5$  bulk targets using  $\lambda=193$  nm,  $T$  substrate  $=375^\circ\text{C}$ , and  $P=100$  mTorr Ar. The values shown were measured at room temperature on as deposited films.

In this series the highest coercivity of 11.3 kOe was obtained for a pulse rate of 10 Hz. The hysteresis loops for films made at laser pulse rates between 10 and 20 Hz were offset in the initial magnetization direction since the maximum applied field of 18 kOe was insufficient to achieve saturation. Lower pulse rates resulted in low coercivities believed to be caused by surface gas contamination due to the longer times between successive pulses.

A key result of this article is that it has been possible to produce  $\text{SmCo}$  films directly by shadowed PLD onto slightly heated substrates with coercivities up to 11.3 kOe at 10 Hz as a function of the laser pulse repetition rate. Higher laser pulse rates did not allow sufficient site relaxation between pulses, while lower pulse rates lead to film contamination because of the longer time between deposition pulses. An operative deposition condition is that  $\sqrt{T/R} = \text{const} \cdot d$ , where  $T$  is the absolute substrate temperature with the surface atom speed proportional to  $\sqrt{T}$ ,  $d$  is the site relaxation distance, and  $1/R$  is the time between laser pulses for the pulse rate  $R$ . Higher laser pulse rates can then only be fruitfully used at higher substrate temperatures where the surface atom mobility is increased to provide site relaxation before the surface is buried by the deposit from the next laser pulse. At higher substrate temperatures the film reactivity with contaminating background gases is expected to increase so that higher substrate temperatures would require lower background gas pressures.

#### ACKNOWLEDGMENTS

The PLD system was purchased with NSF-ARI Grant No. STI-9512308, and with partial support from the NYS Graduate Research Initiative. This work was supported in part by the U.S. Army Research Office, Grant No. DAAH04-94-G-0079, and in part by the Office of Naval Research, Grant No. ONR N00014-96-1-0767. Some support was also derived from the PSC-CUNY Faculty Research Award Program of CUNY.

<sup>1</sup> F. J. Cadieu, H. Hegde, and K. Chen, IEEE Trans. Magn. MAG-25, 3788 (1989).

<sup>2</sup> F. J. Cadieu, "Permanent Magnet Thin Films," in *Physics of Thin Films* (Academic, San Diego, 1992), Vol. 16.

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## Magnetic properties of NdFeB thin films synthesized via laser ablation processing

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High energy product  $\text{Nd}_2\text{Fe}_{14}\text{B}$  films have been grown onto Si(100) substrate by KrF pulsed laser ablation using the targets of  $\text{Nd}_x\text{Fe}_{90.98-x}\text{B}_{9.02}$  ( $x=17.51-27.51$ ). The films exhibit no preferred texture, however, good hard magnetic properties were produced from as-deposited condition:  $4\pi M_s=7$  kG,  $4\pi M_r=4$  kG, and  $H_c=300-1000$  Oe. The beam density of  $3$  J/cm<sup>2</sup> gave the optimal condition to have the highest  $4\pi M_r$  and  $H_c$  for a substrate  $T$  of  $650^\circ\text{C}$ . The higher content of Nd induces a higher coercivity and  $4\pi M_r$  at the same time without prominent change in  $4\pi M_s$ . The films grown at elevated temperature ( $620-700^\circ\text{C}$ ) are understood to suggest a magnetically coupled interaction between  $\text{Nd}_2\text{Fe}_{14}\text{B}$  and  $\alpha\text{-Fe}$  grains by taking into account the monotonously decreasing  $4\pi M_r$  values accompanying with a broad peak regime in  $H_c$  at around  $550-625^\circ\text{C}$ .  
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If high field strength permanent magnets in film forms can be easily synthesized, those film magnets can then be used to fabricate integrated electromagnetic components. Circulators and isolators are the typical hybrid type microwave devices using the ferrite disks for a biasing field. Magnetoresistive heads are another area in which permanent magnetic films can play a significant role to obtain the magnetoresistive sensitivity. Miniaturized magnetic sensors, actuators, and motors are other areas that can benefit from the development of film type magnets.

In spite of these expectations the magnetic field strength of the films must be comparable to the energy product term in bulk magnets. In addition hard magnetic films in microdevices and data storage technology must be cost effective, yet produces smooth hard magnetic films with high values of  $4\pi M_s$ ,  $4\pi M_r$ , and  $H_c$ . Furthermore, a popular film substrate, such as Si wafer, must be proven to be an appropriate material showing no mismatch with the magnetic film materials for volume production in future. Nd-Fe-B compounds of suitable composition and nanostructure is a good candidate for the above requirements. NdFeB magnetic films have been studied during the past years by sputtering a cast target.<sup>1-4</sup> Recently, a high coercive NdFeB films grown on MgO(100) by molecular beam epitaxy (MBE) was reported.<sup>5</sup>

In this article we report a laser ablation technique for synthesizing  $\text{Nd}_2\text{Fe}_{14}\text{B}$  base films using a KrF excimer laser. Our interest is on controlling the microstructure in nanoscale to obtain a high field strength films which presumably will consist of magnetically coupled soft and hard grains.

The magnetic films were prepared by a KrF excimer laser (248 nm wave) ablation. The lasing energy density ranged from  $2.75$  to  $5.99$  J/cm<sup>2</sup> at a constant repetition rate of  $10$  Hz was employed using  $\text{Nd}_x\text{Fe}_{90.98-x}\text{B}_{9.02}$  ( $x=17.5-27.5$ ) targets. The films were deposited onto Si(100) wafer at various substrate temperatures ranging  $620-700^\circ\text{C}$ . Targets were made by induction arc melting and repeated

homogenization treatment. The film thickness was controlled by total pulse of  $2500-3000$  Å for all the samples which exhibited an optimal magnetic properties. The average deposition rate was identified after pulsation for ten minutes for each lasing condition, and the thickness was checked by the scanning electron microscope and "α-Step" as well.

Characterization was performed as functions of various ablation parameters such as laser beam density, Nd contents of targets and substrate temperatures. The film samples were pulsed to have about  $300$  nm thick with a rate of  $10$  Hz. As the lasing beam density increases linearly from  $2.75$  to  $5.99$  J/cm<sup>2</sup>, the deposition rate of film varied linearly from  $0.038$  to  $0.13$  nm/s.

Figure 1 shows the typical magnetic properties as a function of beam energy density. The target composition was

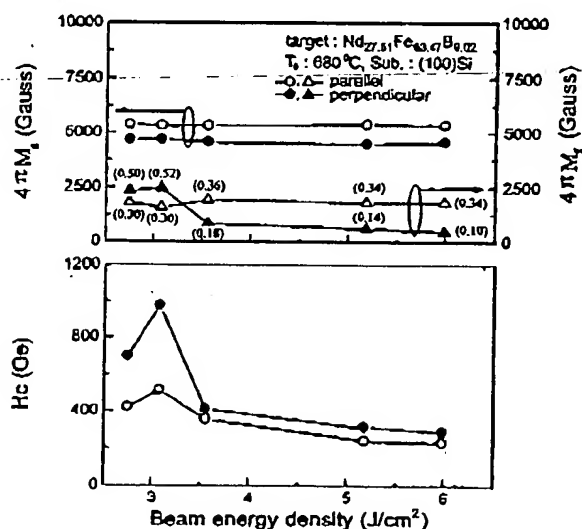


FIG. 1. Laser beam energy dependency on the magnetic properties of  $\text{Nd}_{27.51}\text{Fe}_{63.47}\text{B}_{9.02}$  film on  $(100)\text{Si}$  deposited at a substrate temperature of  $680^\circ\text{C}$ .

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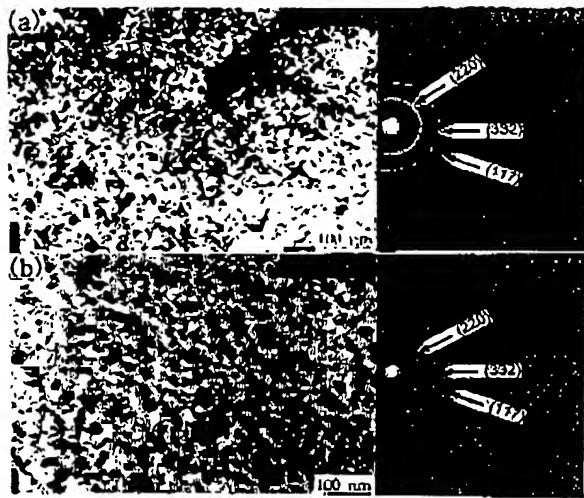


FIG. 2. TEM micrographs and electron diffraction patterns of  $\text{Nd}_{27.51}\text{Fe}_{63.47}\text{B}_{9.02}$  films deposited at a laser beam energy density of (a)  $3.08 \text{ J/cm}^2$  and (b)  $5.99 \text{ J/cm}^2$ , respectively, at  $680^\circ\text{C}$ .

$\text{Nd}_{27.51}\text{Fe}_{63.47}\text{B}_{9.02}$ , and the film was grown at  $680^\circ\text{C}$ . The open circles denote data measured along the in-plane, and the solid circles for out-of-plane direction. The experimentally saturated magnetic moments ( $4\pi M_s$ ) along parallel direction ranged about 5300–5700 G, while those of 1000 G lower values were exhibited along the out-of-plane direction. Saturation magnetization was found to be almost constant regardless of the laser energy density. However, remanence values along the out-of-plane were measured to be much higher for laser energy density  $2.75\text{--}3.08 \text{ J/cm}^2$  as shown in Fig. 1(b). The numbers in round brackets, which are reduced remanence ( $M_r/M_s$ ) values, for the samples grown with the laser energy density of  $2.75\text{--}3.08 \text{ J/cm}^2$  are at most  $0.5\text{--}0.52$ . The laser energy density higher than  $3.5 \text{ J/cm}^2$  gave poor shape of hysteresis curves resulted in low values of both the  $4\pi M_r$  and  $H_c$ . Although the magnetic properties of the films decrease with higher beam densities, the microstructure of all films indicated the presence of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  without any minor phase clarified by x-ray diffraction (XRD) and TEM. Only the difference in the microstructure between the films at lower ( $3.08 \text{ J/cm}^2$ ) and higher beam density ( $6.0 \text{ J/cm}^2$ ) is the grain size as shown in Fig. 2. The grain size of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  phase of the film at a higher beam density ( $6.0 \text{ J/cm}^2$ ) is measured to be doubled (36 nm) that of film at  $3.08 \text{ J/cm}^2$ .

Figure 3 shows the variation of magnetic properties as a function of substrate temperature ( $T_s$ ) for the films grown at a laser beam density of  $3.08 \text{ J/cm}^2$ . For  $T_s < 640^\circ\text{C}$ , the hysteresis behaves like a soft magnet which shows  $4\pi M_r \leq 330 \text{ G}$  and  $H_c \leq 180 \text{ Oe}$ . If  $T_s$  is raised above  $640^\circ\text{C}$ , however, both the  $4\pi M_r$  and  $H_c$  increase resulting from a very coercive hysteresis curve, and the  $M_r/M_s$  becomes more than  $0.5$ . X-ray pattern indicated that  $T_s$  below  $640^\circ\text{C}$  let the films largely amorphous without any minor phase. One interesting thing regarding the substrate temperature is that the

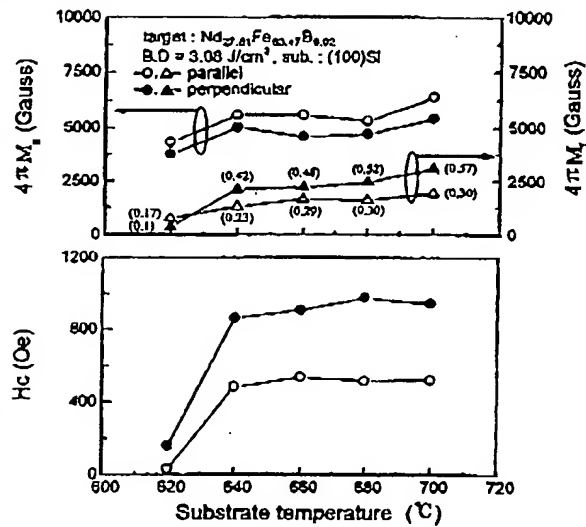


FIG. 3. Substrate temperature dependency on the magnetic properties of  $\text{Nd}_{27.51}\text{Fe}_{63.47}\text{B}_{9.02}$  film on (100)Si deposited at a laser beam energy density of  $3.08 \text{ J/cm}^2$ .

deposition rate of film was found to be more than  $0.6 \text{ Å/s}$  below  $640^\circ\text{C}$ , while above that temperature the rate was less than  $0.5 \text{ Å/s}$ .

The variation of  $4\pi M_r$  and  $H_c$  depend strongly upon the concentration of Nd of the targets. Figure 4 shows the magnetic properties of  $\text{Nd}_x\text{Fe}_{90.98-x}\text{B}_{9.02}$  films grown at  $T_s$  of  $680^\circ\text{C}$  with the beam density of  $3.08 \text{ J/cm}^2$ . The variation of  $4\pi M_r$  as a function of Nd content is low along the both in-plane and out-of-plane directions. However, the  $4\pi M_r$  and  $H_c$  exhibit different behaviors. The films grown from the targets of  $x=17.51$  and  $22.51$  shows the  $M_r/M_s$  along in-

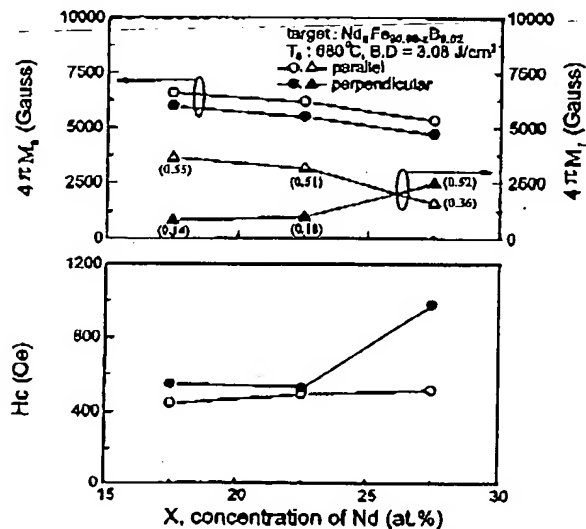


FIG. 4. The effect of Nd content on the magnetic properties of  $\text{Nd}_x\text{Fe}_{90.98-x}\text{B}_{9.02}$  films on (100)Si deposited at the substrate temperature of  $680^\circ\text{C}$  at a laser density of  $3.08 \text{ J/cm}^2$ .

plane of 0.51–0.55, and along out-of-plane of 0.14–0.18 with a low value of  $4\pi M_r$  and  $H_c$  as well. For the films  $x=27.51$ , however, the  $4\pi M_r$  and  $H_c$  along out-of-plane change abruptly to have  $M_r/M_s=0.52$  and  $H_c=900$  Oe, respectively. X-ray diffraction patterns of  $x=17.51$ , 22.51, and 27.51 clearly show that the spectrum of lower Nd contents indicates the poor formation of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  crystallite, while the pattern of  $x=27.51$  shows well developed crystallites. In spite of the variation of Nd content the films formed at  $T_s$  of 620–680 °C consist of single phase of  $\text{Nd}_2\text{Fe}_{14}\text{B}$ , while the films grown at room temperature to indicate the presence of amorphous and crystalline. The previous studies on NdFeB films processed by sputtering<sup>1,3,4,6</sup> exhibited the anisotropic texture aligning the  $c$  axis perpendicular to the film plane.

The high-resolution micrographs of the films  $\text{Nd}_{17.51}\text{Fe}_{73.47}\text{B}_{9.02}$  and  $\text{Nd}_{27.51}\text{Fe}_{63.47}\text{B}_{9.02}$ , respectively, indicate that the basic difference between those Nd concentrations is the grain size. The films of Nd=27.51 at. % shows the finer grains less than 15 nm of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  which resulted in a higher coercivity than those of Nd=17.51 or 22.51 at. % whose grain size was found to be around 20 nm. In the course of ablation around the eutectic temperature some excess of Nd might out out of the alloy, and the formation of Nd-rich grainboundary would take place to anchor the motion of magnetic domains.

Figures 5(a) and 5(b) shows the typical variations of magnetic properties of  $\text{Nd}_{17.51}\text{Fe}_{73.47}\text{B}_{9.02}$  films grown at room temperature and 680 °C, respectively. The data were for the samples measured along in-plane direction taking into account the applicability of the film type permanent magnets exhibiting the high field strength along the in-plane direction of the end devices. In Fig. 5(a) both the  $4\pi M_r$  and  $4\pi M_s$  of as-deposited films are high due to the presence of abundant amorphous phase which also shows the lower coercivity. By post annealing at *in situ* substrate the  $4\pi M_r$  values drop prominently to a certain value (4000 G) and then stabilized thereafter. Almost the same behavior is denoted for  $4\pi M_s$ . As was mentioned above the post annealing seems to induce the fusion of Nd and out out the grain boundaries. However, as the annealing temperature is increased, the  $H_c$  value enhances considerably up to 600 Oe around 600–650 °C. The formation of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  crystallites developed by crystallizing amorphous phase appears to be responsible for the enhancement of  $H_c$ . At higher temperatures,  $H_c$  decreases due to the extended growth of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grains. The above rationalization for the magnetic properties seems to also be true for the films grown at 680 °C in Fig. 5(b).

The high values of  $4\pi M_r$  and  $4\pi M_s$  for the as-deposited films grown at 680 °C tend to decrease with increasing the annealing temperature. At the same time,  $H_c$  values increase to the maximum value (550 Oe) around 550–600 °C and then decrease thereafter [Fig. 5(a)]. In this case, it should be understood that if the film samples exhibit the

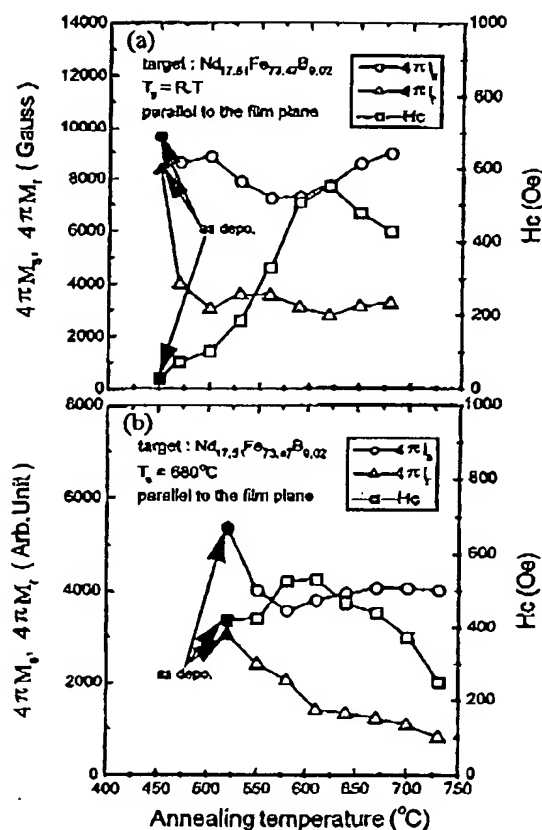


FIG. 5. The variation of magnetic properties as a function of annealing temperature for  $\text{Nd}_{17.51}\text{Fe}_{73.47}\text{B}_{9.02}$  films deposited at (a) room temperature and (b) 680 °C.

same demagnetization effect at each temperature, the decreasing  $4\pi M_r$  should be accompanied with decreasing  $H_c$  values due to the slender hysteresis curve. In addition, the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grains developed during the previous temperature regime will grow, losing their coercivity. However, in Fig. 5(b)  $H_c$  still shows a broad maximum over a small temperature range. These films indicate a coupling interaction between  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grains and magnetically soft  $\alpha\text{-Fe}$  grains which inevitably will form during post annealing.

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ANSI/IEEE Std 100-1984 <sup>2/3</sup>

Third Edition

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## magnetic power factor

518

## magnetic vector

in a nonmagnetic material. 176  
**magnetic power factor.** The cosine of the magnetic hysteresis angle (the sine of the magnetic loss angle). 210

**magnetic recorder.** Equipment incorporating an electromagnetic transducer and means for moving a magnetic recording medium relative to the transducer for recording electric signals as magnetic variations in the medium. *Note:* The generic term magnetic recorder can also be applied to an instrument that has not only facilities for recording electric signals as magnetic variations, but also for converting such magnetic variations back into electric variations. *See:* phonograph pickup. 176

**magnetic recording (facsimile).** Recording by means of a signal-controlled magnetic field. *See:* recording (facsimile). 12

**magnetic recording head.** In magnetic recording, a transducer for converting electric currents into magnetic fields, in order to store the electric signal as a magnetic polarization of the magnetic medium. 176

**magnetic recording medium.** A material usually in the form of a wire, tape, cylinder, disk, etcetera, on which a magnetic signal may be recorded in the form of a pattern of magnetic polarization. 176

**magnetic reproducer.** Equipment incorporating an electromagnetic transducer and means for moving a magnetic recording medium relative to the transducer, for reproducing magnetic signals as electric signals. 176

**magnetic reproducing head.** In magnetic recording, a transducer for collecting the flux due to stored magnetic polarization (the recorded signal) and converting it into an electric voltage. 176

**magnetic rotation (polarized light) (Faraday effect).** When a plane polarized beam of light passes through certain transparent substances along the lines of a strong magnetic field, the plane of polarization of the emergent light is different from that of the incident light. 210

**magnetic sensitivity (Hall-effect devices).** The ratio of the voltage across the Hall terminals to the magnetic flux density for a given magnitude of control current. 107

**magnetic spectrograph.** An electronic device based on the action of a constant magnetic field on the paths of electrons, and used to separate electrons with different velocities. *See:* electron device. 244, 191

**magnetic starter (packaging machinery).** A starter actuated by electromagnetic means. 429

**magnetic storage.** A method of storage that uses the magnetic properties of matter to store data by magnetization of materials such as cores, films, or plates, or of material located on the surfaces of tapes, discs, or drums, etcetera. *See:* magnetic core; magnetic drum; magnetic tape. 235

**magnetic storm.** A disturbance in the earth's magnetic field, associated with abnormal solar activity, and capable of seriously affecting both radio and wire transmission. *See:* radio transmitter. 328

**magnetic susceptibility (Isotropic medium).** In rationalized systems, the relative permeability minus unity.

lized systems, the relative permeability minus unity.

$$k = \mu_r - 1 = B_i / \mu_0 H$$

*Notes:* (1) In unrationalized systems,  $k = (\mu_r - 1)4\pi$ . (2) The susceptibility divided by the density of a body is called the susceptibility per unit mass, or simply the mass susceptibility. The symbol is  $\chi$ . Thus

$$\chi = k / \rho$$

where  $\rho$  is the density.  $\chi$  multiplied by the atomic weight is called the atomic susceptibility. The symbol is  $\chi_A$ . (3) In anisotropic media, susceptibility becomes a matrix. 210

**magnetic tape (homogeneous or coated).** (1) A tape with a magnetic surface on which data can be stored by selective polarization of portions of the surface. (2) A tape of magnetic material used as the constituent in some forms of magnetic cores. *See:* coated magnetic tape. 235, 255, 77, 54

**magnetic tape handler (test, measurement and diagnostic equipment).** A device which handles magnetic tape and usually consists of a tape transport and magnetic tape reader with associated electrical and electronic equipments. Most units provide for tape to be wound and stored on reels; however, some units provide for the tape to be stored loosely in closed bins. 54

**magnetic tape reader (test, measurement and diagnostic equipment).** A device capable of converting information from magnetic tape where it has been stored as variations in magnetizations into a series of electrical impulses.

**magnetic test coil (search coil) (exploring coil).** A coil that, when connected to a suitable device, can be used to measure a change in the value of magnetic flux linked with it. *Note:* The change in the flux linkage may be produced by a movement of the coil or by a variation in the magnitude of the flux. Test coils used to measure magnetic induction  $B$  are often called  $B$  coils; those used to determine magnetizing force  $H$  may be called  $H$  coils. A coil arranged to rotate through an angle of 180 degrees about an axis of symmetry perpendicular to its magnetic axis is sometimes called a flip coil. *See:* magnetometer. 328

**magnetic thin film.** A layer of magnetic material, usually less than 10 000 angstroms thick. *Note:* In electronic computers, magnetic thin films may be used for logic or storage elements. *See:* coated magnetic tape; magnetic core; magnetic tape. 235, 77

**magnetic track braking.** A system of braking in which a shoe or slipper is applied to the running rails by magnetic means. *See:* electric braking. 328

**magnetic variometer.** An instrument for measuring differences in a magnetic field with respect to space or time. *Note:* The use of variometer to designate a continuously adjustable inductor is deprecated. *See:* magnetometer. 328

**magnetic vector (radio wave propagation).** *See:* magnetic field vector. 146

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Note

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